

(43) Date of A Publication 28.03.2001

(21) Application No 0022911.2

(22) Date of Filing 19.09.2000

(30) Priority Data

(31) 9912266

(32) 21.09.1999

(33) FR

(71) Applicant(s)

Institut Francais du Petrole  
(Incorporated in France)  
1 & 4 Avenue de Bois-Preau,  
92852 Rueil-Malmaison Cedex, France

(72) Inventor(s)

Elodie Goglin  
Marc Fleury  
Patrick Egermann

(74) Agent and/or Address for Service

Fitzpatricks  
39 Stukeley Street, LONDON, WC2B 5LT,  
United Kingdom

(51) INT CL<sup>7</sup>

G01N 15/08 33/24

(52) UK CL (Edition S )

G1S SGX

(56) Documents Cited

GB 2332282 A GB 2325526 A US 5463894 A

(58) Field of Search

UK CL (Edition R ) G1S SGX S1A  
INT CL<sup>7</sup> G01N 13/00 15/08 33/24  
On-line: EPODOC, WPI, JAPIO

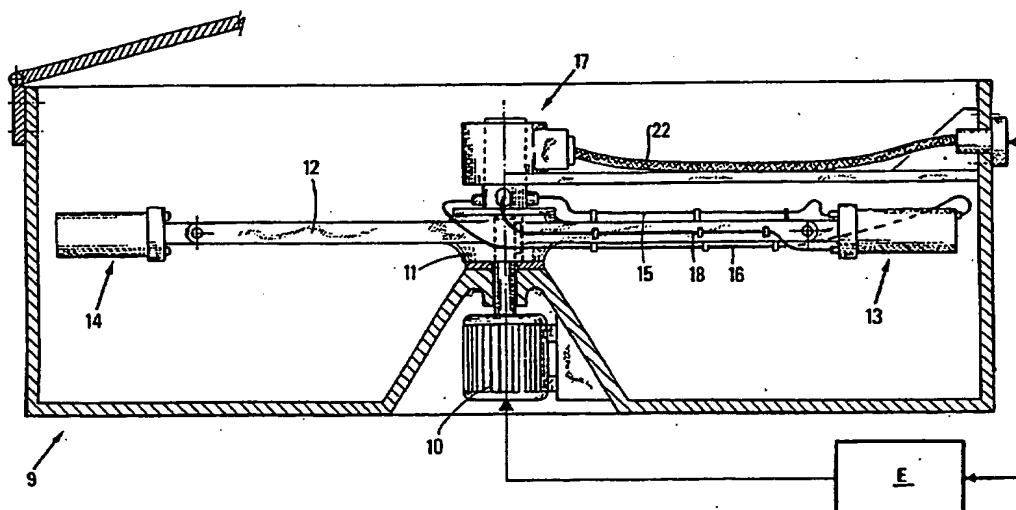
(54) Abstract Title

**A method, including modelling, for determining physical parameters of a sample**

(57) Method for faster determining certain physical parameters (capillary pressure curves and relative permeabilities for example) of a geologic sample initially saturated with a first fluid placed in a vessel and subjected to centrifugation in the presence of a second fluid. The equilibrium saturations are determined long before complete stabilization is reached, through analytic modelling involving exponential functions, best adjusting to an effective production curve, for each speed stage. A database obtained by means of various simulations is used to obtain the relative permeability curves corresponding to the centrifuged sample. The method can be applied for measurement of petrophysical characteristics.

The method comprises measuring the amount of fluids displaced by centrifugation and establishing saturation curves, characterized in that it comprises, for each rotating speed, modelling a saturation curve best corresponding at each time interval to the curve portion already obtained. The figure shows a centrifugation device having a cup 13 for the porous rock sample, which is initially saturated with brine and placed in cup 13. The cup 13 is initially filled with an electrically insulating fluid such as oil.

FIG.1

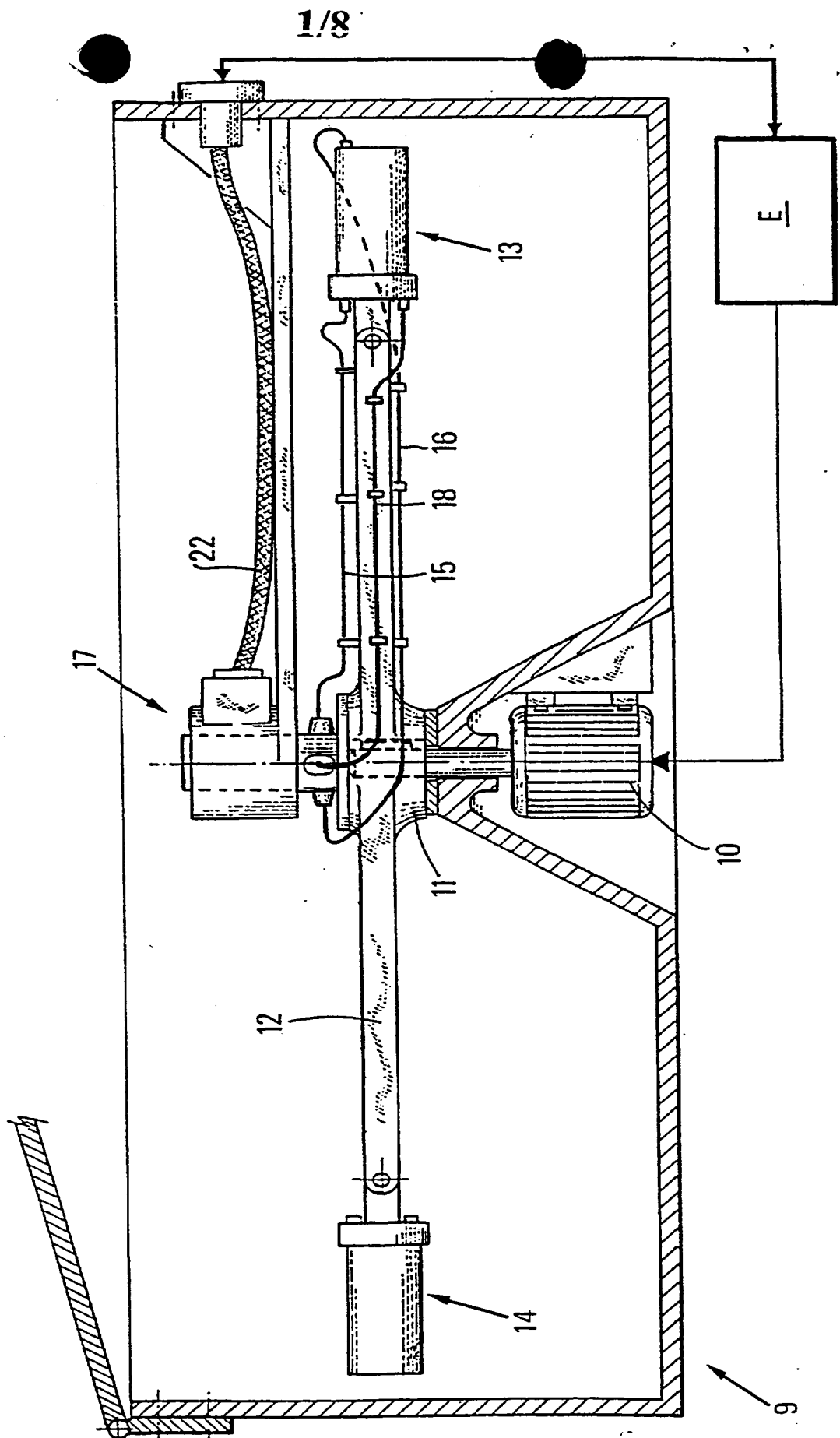


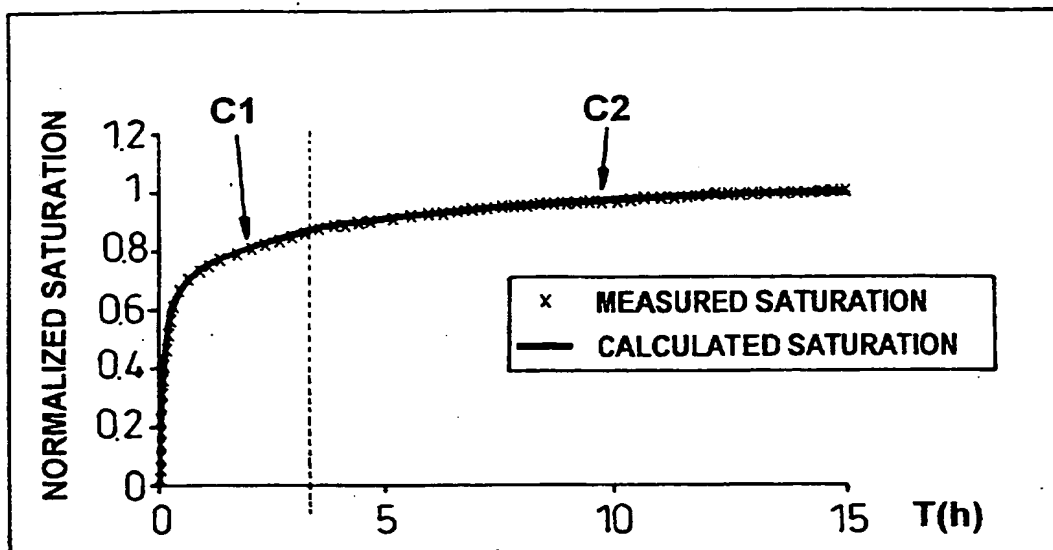
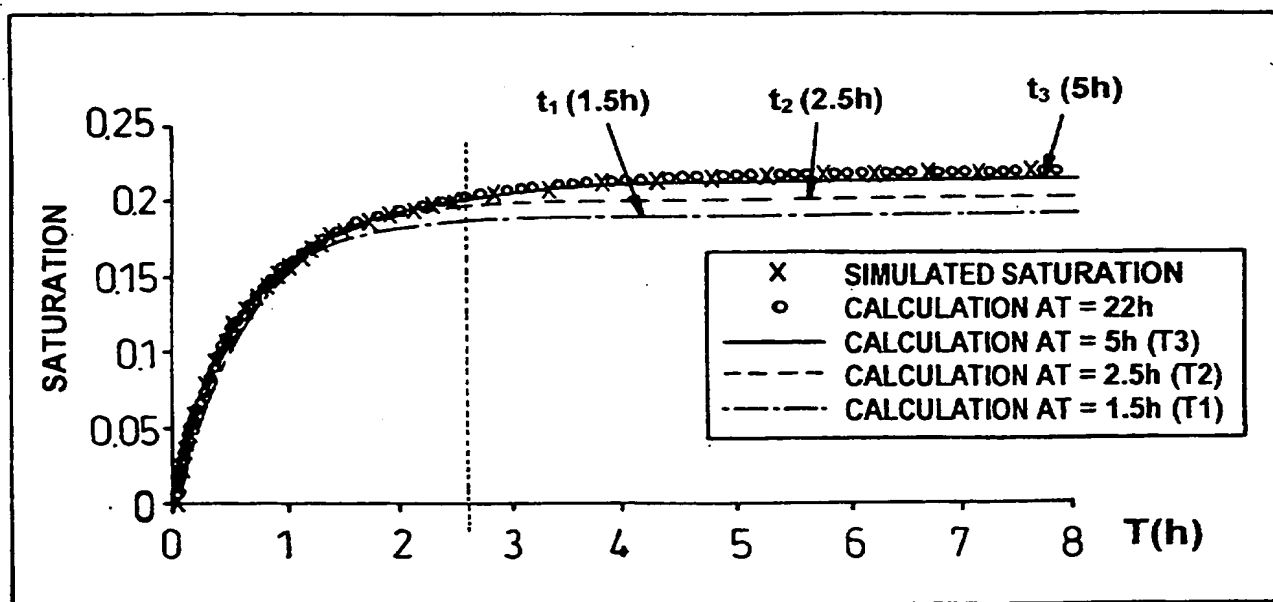
At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

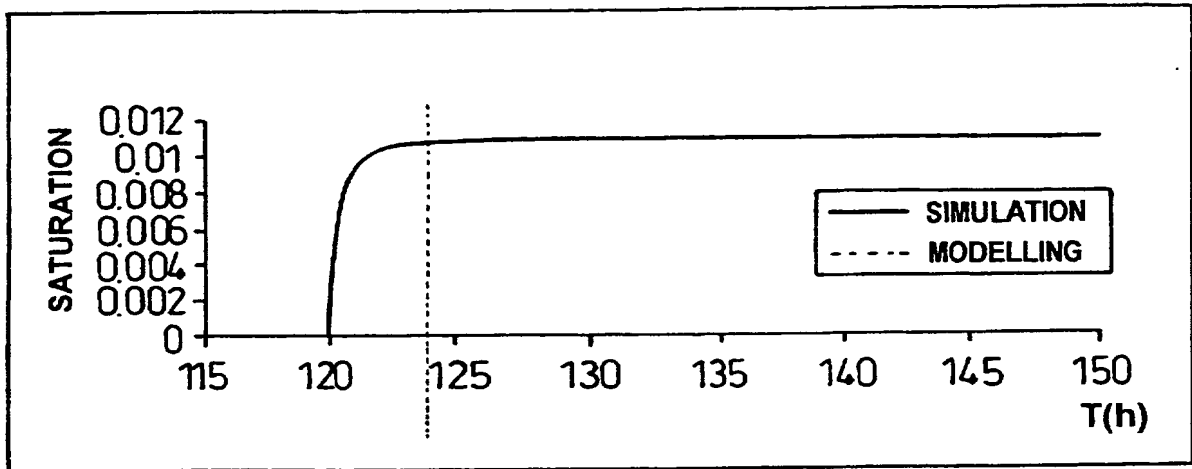
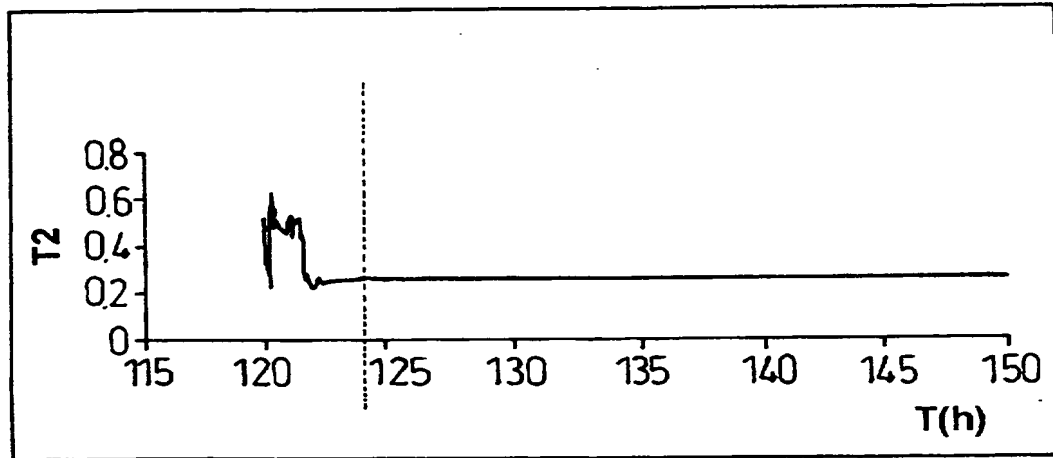
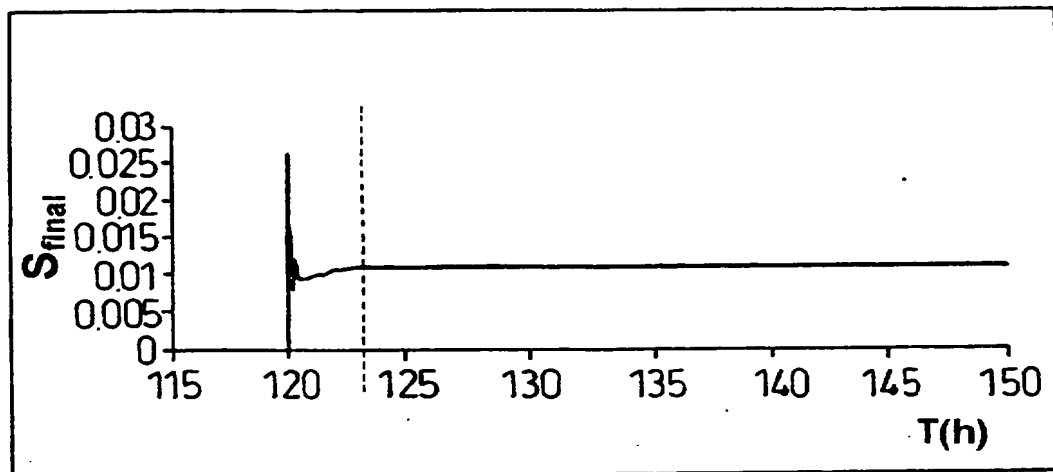
BEST AVAILABLE COPY

GB 2 354 590 A

FIG.1



*Fig. 2**Fig. 3*

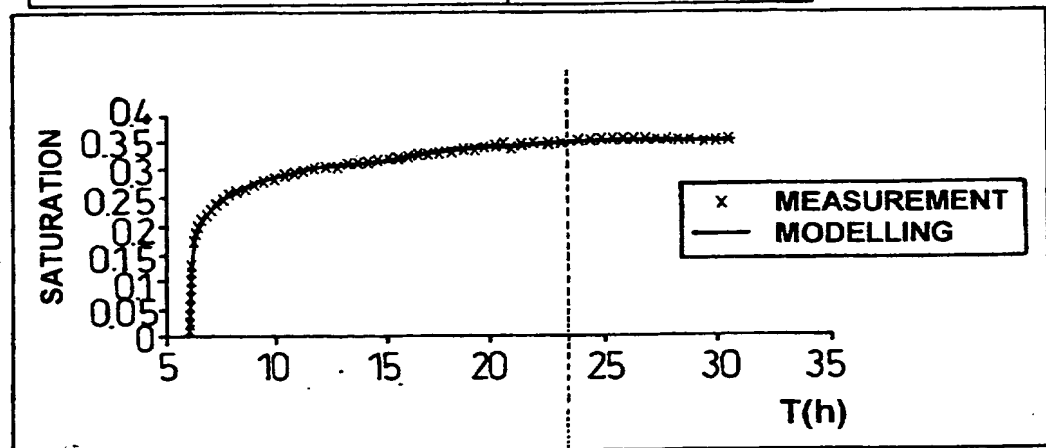
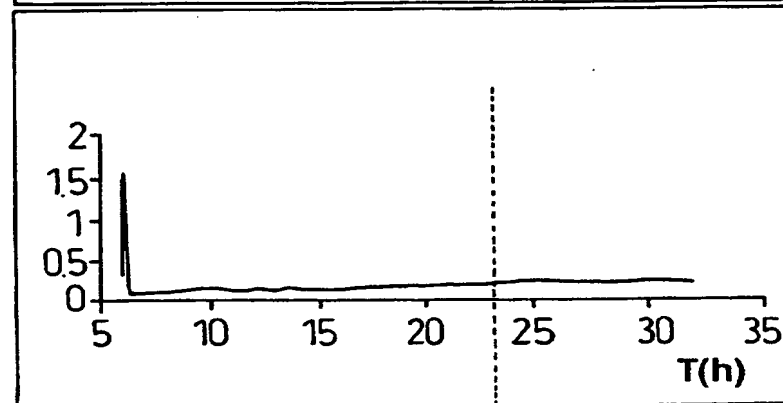
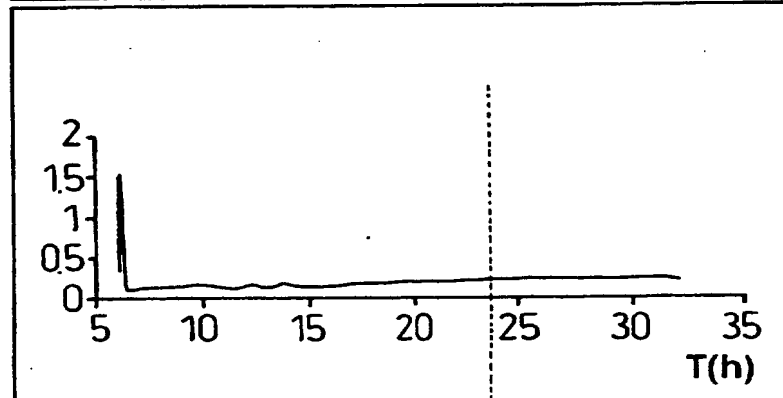
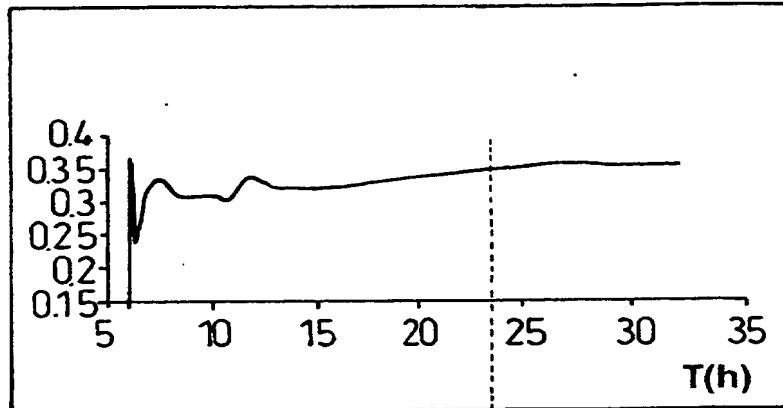
*Fig. 4A**Fig. 4B**Fig. 4C*

Time (h)	101.6	122.04	123	124.56	150.24
$T_2 (h^{-1})$		0.21752	0.24931	0.25233	0.24255
$S_{final}$		0.01064	0.01074	0.01080	0.01084
ERROR ON $T_2$ (%)		10.44248	2.64543	3.88978	0.13713
ERROR ON $S_{final}$ (%)		1.78773	0.92107	0.40525	0.00972

**Fig. 5**

Time (h)	3.07806	3.57805556	5.15722	5.84111
$T_2 (h^{-1})$	1.45842	1.81289	1.55651	1.60005
$S_{final}$	0.97379	1.00749	0.99701	1.00092
ERROR ON $T_2$ (%)	8.85142	13.3027	2.72111	0.00000
ERROR ON $S_{final}$ (%)	2.71030	0.65730	0.39043	0.00000

**Fig. 7**



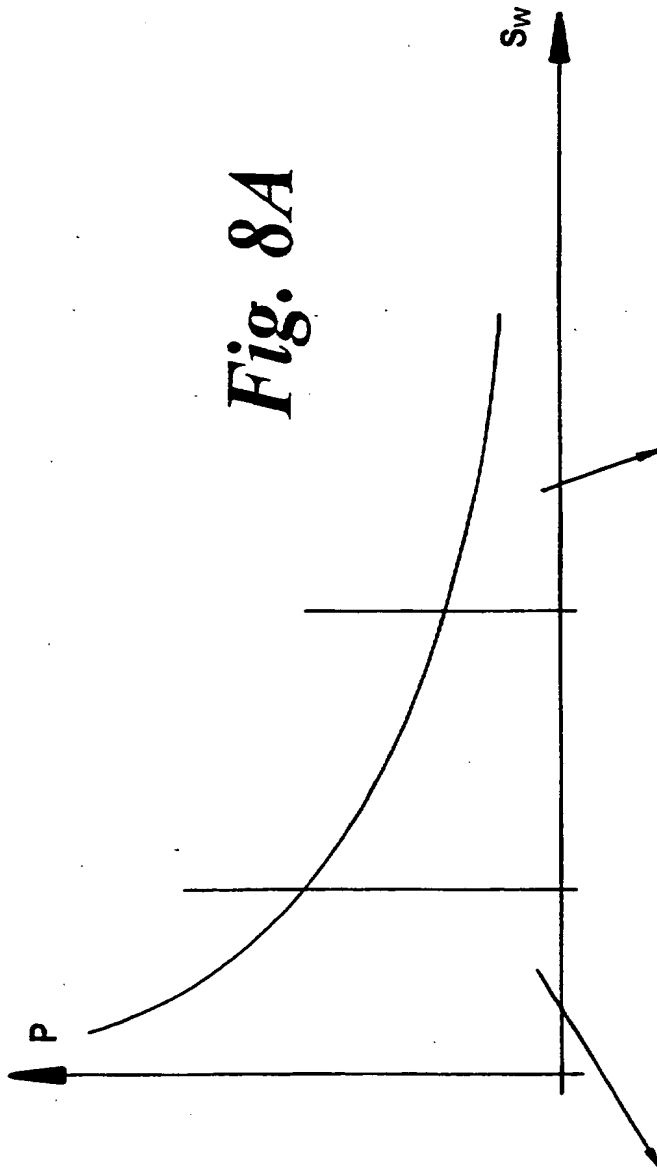


Fig. 8A

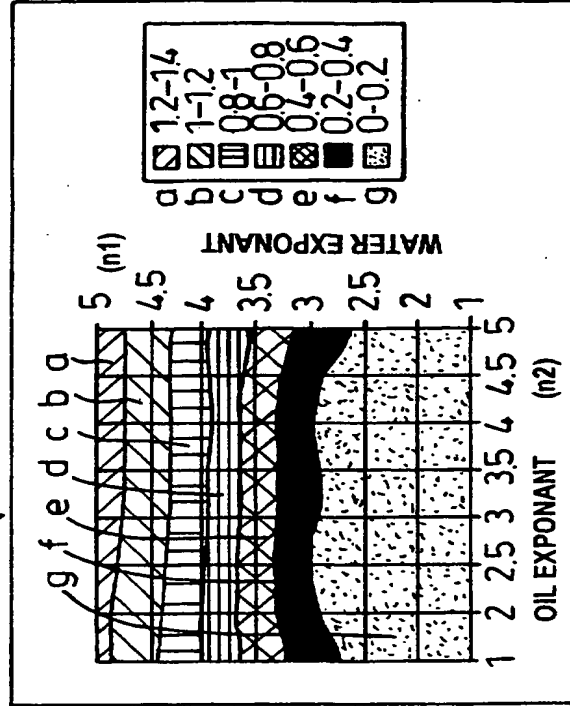


Fig. 8C

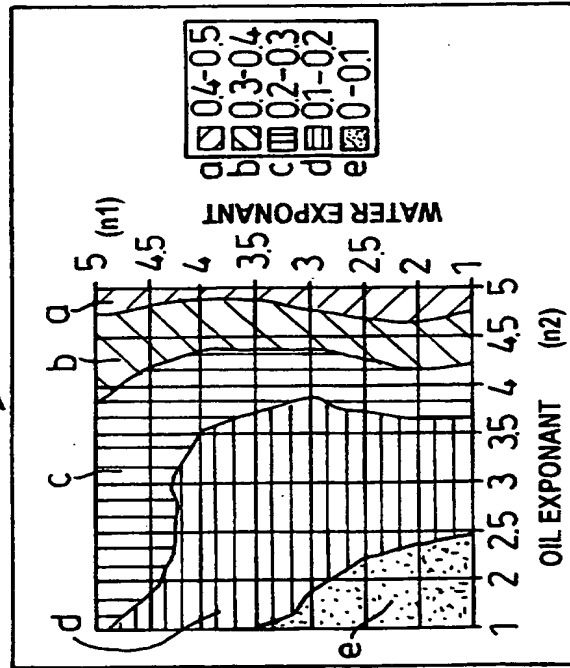
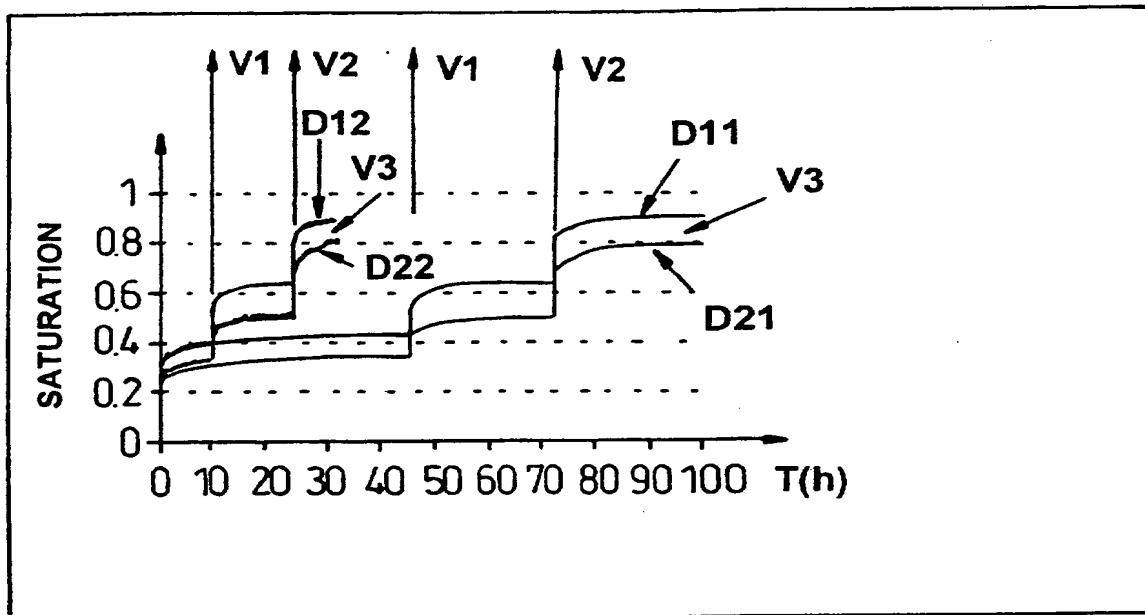
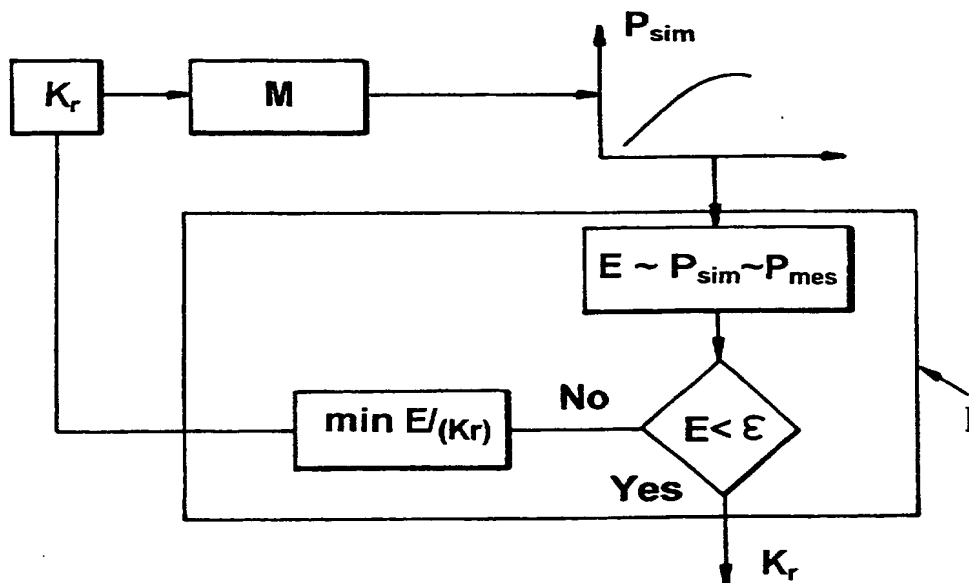


Fig. 8B



**Fig. 9**



**Fig. 11**



	CONVENTIONAL DRAINAGE		FAST DRAINAGE		ERROR (%)	
	1	2	1	2	1	2
V1	$S_{final}$	0.345	0.424	0.331	0.401	4.03
	DURATION	45	10		GAIN	78%
	ACCURACY	2.586E-05	1.711E-05	1.404E-06	1.761E-06	
V2	$S_{final}$	0.502	0.632	0.508	0.636	1.10
	DURATION	25	15		GAIN	40%
	ACCURACY	5.193E-06	3.481E-06	1.341E-05	1.403E-05	
V3	$S_{final}$	0.795	0.908	0.827	0.897	3.95
	DURATION	25	6		GAIN	75%
	ACCURACY	1.535E-05	7.565E-06	1.628E-06	3.767E-07	

Fig. 10

5       The method finds interesting applications in the field of petrophysics where knowledge of parameters such as the capillary pressure, the relative permeability curves of porous rocks, allows reservoir engineers to characterize the flow conditions of hydrocarbons in a reservoir, to make production predictions and to elaborate development strategies.

10 BACKGROUND OF THE INVENTION

A well-known method for rapidly determining both the capillary pressure curves and the relative permeability curves of a porous rock sample initially saturated with a first fluid essentially consists in subjecting it to centrifugation in order to displace the fluid in place by means of another fluid.

15 Centrifugation devices such as those described in patents FR-2,603,040 (US-5,463,894), FR-2,763,690, FR-2,772,477 filed by the applicant are for example used.

The sample initially saturated with a liquid A (brine for example) is placed in an elongate vessel or cup containing another fluid B of different density (oil for example). The vessel is fastened to the end of a rotating arm and a centrifugal force is applied thereto so as to study the displacements of the fluids in the sample during at least two distinct stages. During a first drainage stage, the assembly is subjected to a centrifugal force exerted along the length of the vessel so as to exert thereon an expulsion force

which tends to cause part of first fluid A to flow out. Simultaneously, part of fluid A penetrates the sample. The two fluids move in the sample to a position of equilibrium where the force due to the capillary pressure in the pores makes up for the centrifugal force exerted. A measuring sonde is placed in the vessel, on the side of the sample. The sonde can be a capacitive type sonde for example, and it can comprise a metal rod insulated by means of a ceramic sheath. The capacitance between the rod and the conducting fluid (brine), which is proportional to the height immersed, is measured. With this system, the measuring accuracy is 1.5 % of the pore volume. The sonde detects the position of the interface between the two liquids in the vessel and transmits the measurement signals to a signal acquisition and control device comprising hydraulic liquid circulation means and an acquisition device.

The various points of the capillary pressure curve ( $P_c$ ) are obtained from measurement of the mean saturation of the sample at equilibrium for given rotating speeds. Determination of the relative permeability curves ( $K_r$ ) is achieved by means of the saturation variation with time between two (transient) speed stages. None of these curves is obtained directly ; the first one must be calculated and the second one requires a numerical flow simulator comprising (Fig.11) a simulation module which reproduces the effects of the experiment by means of a numerical model, and an inversion module allowing to determine capillary pressure and relative permeability curves by iterative adjustment of an initial numerical model to the experimental results.

An example of a well-known flow simulator is the ACEX flow simulator based on Darcy's generalized equations, which is described in :

- Chardaire-Rivière, C. ; 1992, Improving the Centrifuge Technique by Measuring Local Saturations ; SPE 24882.

Although the centrifugation technique is widely used in the petroleum industry, because of its relative rapidity in relation to other existing methods, some implementation difficulties remain concerning determination of the capillary pressure curve as well as the relative permeability curves.

5 For a given speed stage, the transient state is difficult to determine since a very slow production stabilization is observed. Search for this equilibrium leads to long-lasting operations spread over several weeks, whose implementation presents practical difficulties, considering especially the often high rotating speed of the centrifuge. Furthermore, the time required therefore is even longer if one tries to increase the pore  
10 volume of the sample (by increasing the length thereof) in order to improve the saturation measuring accuracy.

Inversion, i.e. adjustment of a numerical model to experimental results, is difficult to achieve because the information relating to the relative permeabilities is concentrated on the transient part of the production curve, i.e. in a narrow range of experimental  
15 values. The acceleration applied to the centrifuge has a preponderant effect on the form of this transient part, notably its duration. Parallel determination of the capillary pressure curve leads to applying several constant speed stages rather than a long acceleration, which reduces the transient curves.

### SUMMARY OF THE INVENTION

20 The modelling method according to the invention allows to obtain, with a considerable gain in time, the values of physical parameters of a geologic sample initially saturated with a first fluid placed in a vessel and subjected to centrifugation in the presence of a second fluid.

It comprises measuring the amount of fluids displaced by centrifugation and establishing saturation curves, at fixed time intervals, and it is characterized in that it comprises, for each rotating speed stage, modelling of a saturation curve best adjusting, at each time interval, to the curve portion already obtained and corresponding to the various previous measurements, the value of the saturation at equilibrium being deduced from characteristics of the modelled curve.

For each speed stage, an exponential type saturation curve is modelled by adjusting parameters, one of these parameters corresponding to the value of the saturation at equilibrium. The saturation curve is modelled for example by means of a sum of exponential functions depending on said adjustment parameters.

Centrifugation speeds allowing to obtain an even distribution of points on each saturation curve are preferably selected.

According to an embodiment, the method further comprises :

- using a flow simulator for modelling a relation between the relative permeability values in relation to the two fluids for a sample of determined dimensions and permeability, and the saturation values in relation to these fluids according to various coefficients, and establishing a database connecting parameters intended for adjustment to said coefficients, for several saturation values in relation to at least one of the fluids, and
- determining relative permeabilities of the sample subjected to centrifugation with reference to correspondence values extracted from the database.

## BRIEF DESCRIPTION OF THE DRAWINGS

Other features and advantages of the method according to the invention will be clear from reading the description hereafter of a non limitative example, with reference to the accompanying drawings wherein :

- 5    - Figure 1 shows a centrifugation device allowing the method to be implemented,
- Figure 2 shows an example of adjustment of a modelling curve to an experimental production curve,
- Figure 3 shows, by way of comparison, various modelling curves obtained at different times of the same speed stage,
- 10   - Figures 4a, 4b, 4c show the compared evolutions respectively of a production curve, a variation curve of the time constant  $T_2$  involved in modelling of the simulation curve, and the variation curve of the final value  $S_{\text{final}}$  that can be predicted by simulation,
- Figure 5 is a table showing an example of compared evolutions of several parameters during the same speed stage,
- 15   - Figures 6a, 6b, 6c, 6d respectively show the evolutions in time of the saturation value  $S_{\text{final}}$  of a sample, of coefficients  $T_1$ ,  $T_2$  used as parameters in the simulated curve and the corresponding production curve,
- Figure 7 is a table showing a second example of compared evolutions of several parameters during the same speed stage,
- 20   - Figure 8a diagrammatically shows a saturation variation curve  $S_w$  and Figures 8b and 8c respectively show graphs connecting various coefficients and which correspond to two saturation variation zones,

- Figure 9 shows the compared evolutions of saturation curves obtained according to the conventional method (D11, D21) and according to the fast method according to the invention (D12, D22),

- Figure 10 is a comparative table of numerical values for three speed stages, according to whether the conventional method or the accelerated method according to the invention is used, and

- Figure 11 diagrammatically shows the organization of a flow simulator.

### DETAILED DESCRIPTION

A centrifugation device as described hereafter is used for implementing the method.

10 It comprises (Fig.1) an elongate vessel or cup 13 intended for a sample. A capacitive type level-detecting sonde (not shown) is arranged in a tubular lateral chamber parallel to the direction of elongation of the sample and of the vessel. Any variation in the level of the conducting fluid in the vessel leads to a capacitance variation of the sonde. Centrifuge 11 comprises a tank 9, an electric motor 10 whose  
15 shaft drives a hub 11 in rotation. Two identical arms 12 are mounted in opposition on hub 11. Two vessels or cups 13, 14 are mounted swivelling at the ends of arms 12 so as to spontaneously line up along the direction of the centrifugal force applied, and they balance each other in rotation. A porous rock sample to be measured, initially saturated with an electrically conducting fluid A, such as brine for example, is placed in vessel  
20 13. Vessel 13 is initially filled with an electrically insulating other fluid B such as oil.

Two lines 15, 16 fastened along one of arms 12 connect vessel 13 to a rotary electro-hydraulic connector 17 carried by hub 11. First line 15 is used for injecting oil

into vessel 13. The second one, 16, is used for collecting the brine drained out of the sample by the rotation. A cable 18 connects the measuring sonde to rotary connector 17. By means of connector 17, the conductors of cable 18 are electrically connected to another cable 21. These two lines and this cable form a linking element 22 between vessel 13 and an external assembly E suited to collect the signals coming from the sonde, to process the acquired data, to control fluid transfers and to actuate driving motor 10.

The method comprises first selecting a certain number of rotating speed stages selected to obtain well-distributed points on the saturation curve. The operator can select them by referring to values obtained with previous tests for samples of the same type. Failing that, he can also quickly carry out a test on a sample by means of a Purcell type method well-known to specialists, so as to establish a corresponding capillary pressure curve and, according to the form thereof, to estimate the suitable speed stages.

The fast access to the saturation values obtained with the method is based on analytic modelling of the fluid production stages of the sample subjected to centrifugation. An exponential type modelling equation is used therefore.

It has been found that most production curves can be modelled by the following equation :

$$S(t) = S_{eq} [1 - (P \cdot e^{-t/T_1} + (1-P) \cdot e^{-t/T_2})] + S_{ini}$$

where  $S_{eq}$  is the saturation at equilibrium,  $S_{ini}$  the initial saturation of the previous speed stage,  $t$  the time,  $P$  a weighting factor, and  $T_1$ ,  $T_2$  are time constants.



The lowest time constant  $T1$  describes the transient part  $C1$  of the saturation curve (Fig.2), i.e. the dynamic effects. The highest time constant  $T2$  describes stabilization zone  $C2$ , i.e. capillary effects.

In order to estimate the saturation at equilibrium, long before the stage where its variations actually become lower than the measuring accuracy, coefficients  $T1$ ,  $T2$  are sought which, applied in the equation above, generate a saturation curve that adjusts to the part of the production curve that is already known.

At each new rotating speed stage, the control device performs a new measurement acquisition and a new pair of data is obtained (time, production). The programme then adjusts the bi-exponential mathematical modelling to the points curve already available (Fig.3). The evolution of time constant  $T2$  is thus obtained, which determines the stabilization and final saturation  $S_{final}$  with time. In this programme, a maximum difference is fixed between two consecutive values of  $T2$  and  $S_{final}$ , corresponding for example to the practical precision of the measuring device. When the calculated differences are below these values, it may be assumed that  $T2$  and  $S_{final}$  are sufficiently stabilized and therefore that the production curve is entirely determined, and the next speed stage can be dealt with.

Their evolution is shown in Figs.4a to 4c. A very marked stabilization of time constant  $T2$  and of the final saturation can be observed. This stabilization occurs as soon as the flow reaches the final plateau, i.e. as soon as the capillary forces become preponderant. Constant  $T1$  stabilizes much faster since it only relates to the first part of the curve.

The table in Figure 5 shows the evolution with time of four parameter sets P, T1, T2,  $S_{\text{final}}$  calculated at four different times of the experiment.

If the accuracy of the capacitive sonde measuring saturation S in the vessel containing the sample is of the order of 1.5 % of the pore volume for example, i.e. 1.5 % in saturation, it can be considered that the final saturation value is known after a 3-hour experiment. This implies a 2.6 % error on T2 and a 0.9 % error on  $S_{\text{final}}$ , below the accuracy of the sonde. If one waited for effective stabilization of the saturation where the differences between successive measurements become smaller than the sonde accuracy, the speed stage should last about ten hours. The duration of the experiment is thus divided by three.

The peak, then the stabilization observed on the simulated curves are found for parameters T2 and  $S_{\text{final}}$  from an experimental curve. The evolutions shown in Figs.6a to .6d are obtained.

The table in Figure 7 shows similar results for another experimental case.

After 3.5 hours, the final saturation value is known to 0.66 %, which is less than the measuring accuracy. If one had waited for stabilization, the experiment would have lasted for more than 7 hours. The duration of the test can thus be reduced by half, by a factor of the order of 2 or 3. It can thus be considered that, considering these two examples, when the value of T2 is stable to 5 % and the value of  $S_{\text{final}}$  is stable to 1 %, the mathematical modelling parameters are sufficiently determined to allow the experiment to be stopped and the next speed stage to be dealt with.

### Determination of the relative permeability curves

Corey's equation, well-known to specialists, is used for determining the relative permeability curves for oil and water,  $K_{r,nr}$  and  $K_{r,r}$  respectively, from the respective saturations  $S_{nr}$  and  $S_r$  obtained during the experiment :

$$\frac{K_{r,r}}{K_{r,r}^0} = S_r^{n_1} \text{ and } \frac{K_{r,nr}}{K_{r,nr}^0} = S_{nr}^{n_2} \quad (2)$$

5 where  $n_1, n_2$  are coefficients.

A database is therefore formed, that can be presented in form of graphs for example, allowing to connect the coefficients of constant  $T_2$  to exponents  $n_1, n_2$ , for different values of these exponents. A sample of determined length (6 cm for example) and with a given permeability (100 mD for example) is considered, and coefficients  $n_1, n_2$  are  
10 varied between 1 and 5 each.

Production curves  $S(t)$  are determined for various speed stages by means of the simulator. As mentioned above, the values of  $P, T_1, T_2$  allowing best adjustment of the modelled curve to the saturation curve variation are determined by applying relation 1. For all the value triplets  $(T_2, n_1, n_2)$ , value charts are formed or, more practically,  
15 graphs such as those shown in Figs.8a, 8b, corresponding to different stages of each saturation curve (Fig.8c).

For a speed stage  $V_1$  corresponding to a high water saturation  $S_w$ , it can be actually observed that exponent  $n_2$  for oil has a predominant influence. The greater it is, i.e. the more the oil relative permeability is curved, the greater time constant  $T_2$ . It can  
20 however be noted that, for this stage, the water exponent also plays a part, even though it is less marked.

For speed stage V3 corresponding to a low water saturation  $S_w$ , the predominance of exponent  $n_1$  for water is very clear. Exponent  $n_2$  for oil has no influence any longer.

For intermediate stages, it can be seen that the two exponents are by no means insignificant. At this stage, the flow is sensitive to the low relative permeability to each  
5 fluid.

The tables or graphs formed can be used for samples of different lengths and permeabilities, certain corrective factors being then applied.

Using short samples allows to reduce the duration of a production stage. However, using a longer sample allows to spread the transient parts over the curves and thus to  
10 optimize determination of the relative permeabilities. It is observed that, for samples of the same rock but of different lengths, a second degree polynomial type relation can be drawn between the stabilization time and the length. To go from a sample of length  $L_1$  (the sample used for drawing the graphs) to a sample of different length  $L_2$ , the following relation can be applied :

$$\frac{T_2(L_2)}{T_2(L_1)} = \left( \frac{L_2}{L_1} \right)^2$$

15 A quasi-linear evolution of time constants  $T_1$  and  $T_2$  is observed as a function of the absolute permeability of the sample. A corrective factor is therefore applied :

$$\frac{T_2(K_2)}{T_2(K_1)} = \left( \frac{K_2}{K_1} \right)$$

### Validation of the method

Two samples were centrifuged, the first one water wet, the second one rather oil wet, a first time according to the conventional method, the second time according to the method described. 3 successive speed stages were applied : 470, 600 and 1050 rpm, determined after simulation tests, and the production curves shown in Figs.9a, 9b were obtained.

Concerning equilibrium saturations, the results obtained are given in the table of Fig.10.

It can be noted that the final saturation accuracy greatly depends on the duration of the stage.

The fast drainage for stages 1 and 3 thus leads to a final saturation that is slightly different from the effective stabilization. However, the duration of these two stages is greatly reduced. Stage 3 on the other hand gives quite satisfactory results, but its duration is much less reduced.

There is thus a compromise between measuring accuracy and duration. After different tests, it is suggested to fix the variation criteria of parameters  $T_2$  and  $S_{\text{final}}$  to 2 % and 0.5 % respectively. However, these values should be estimated according to each case. In fact, it can be noted that there are samples for which time constant  $T_2$  is difficult to stabilize when the final saturation is already well-determined.

Furthermore, the fact of not waiting for stabilization of the fluids before proceeding to a new stage does not disturb the shape of the production curve. In fact, a bi-exponential modelling can always be associated therewith. The precision of the

modelling adjustment to the experimental curves remains very acceptable (of the order of  $10^{-5}$ ).

A modelling type involving exponential functions has been described. Other types of functions (splines for example) that can be connected to significant physical parameters of the sample could however be used without departing from the scope of the invention.

## CLAIMS

1) A method for faster determining certain physical parameters of a geologic sample initially saturated with a first fluid placed in a vessel and subjected to centrifugation in the presence of a second fluid at speeds varying progressively in stages, comprising measuring the amount of fluids displaced by centrifugation and establishing saturation curves, characterized in that it comprises, for each rotating speed stage, modelling a saturation curve best corresponding at each time interval to the curve portion already obtained, corresponding to various measurements already performed, by selecting adjustment parameters, the value of each final saturation ( $S_{\text{final}}$ ) being deduced from characteristics of the modelled curve.

2) A method as claimed in claim 1, characterized in that an exponential type saturation curve is modelled for each speed stage by adjustment of parameters ( $S_{\text{equ}}$ , P, T), one of these parameters corresponding to the saturation value at equilibrium.

3) A method as claimed in claim 2, characterized in that the saturation curve is modelled by means of a sum of exponential functions depending on said adjustment parameters ( $S_{\text{equ}}$ , P, T1, T2).

4) A method as claimed in any one of the previous claims, characterized in that it comprises selecting centrifugation speeds allowing an even distribution of points to be obtained on each saturation curve.

5) A method as claimed in any one of the previous claims, characterized in that it comprises :

- using a flow simulator for modelling a relation between the values of relative permeability (K) respectively to the two fluids of a sample of determined dimensions and permeability, and the values of saturation (S) with these fluids according to various coefficients (n1, n2) and establishing a database connecting adjustment parameters (T1, T2) to said coefficients, for several saturation values relative to at least one of the fluids, and
- determining relative permeabilities (K) of the sample subjected to centrifugation with reference to correspondence values extracted from the database.

6) A method substantially as hereinbefore described with reference to the accompanying drawings.





**Application No:** GB 0022911.2  
**Claims searched:** ALL

**Examiner:** Michael Walker  
**Date of search:** 12 January 2001

**Patents Act 1977**  
**Search Report under Section 17**

**Databases searched:**

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.S): G1S (SGX, SIA)

Int Cl (Ed.7): G01N 13/00, 15/08, 33/24

Other: On-line: EPODOC, WPI, JAPIO

**Documents considered to be relevant:**

Category	Identity of document and relevant passage	Relevant to claims
A	GB 2332282 A (INST FRANCAIS DU PETROLE)	
A	GB 2325526 A (INST FRANCAIS DU PETROLE)	
A	US 5463894 (FLEURY et al.)	

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.



**THIS PAGE BLANK (USPTO)**

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☒ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**

**THIS PAGE BLANK (USPTO)**